

(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets



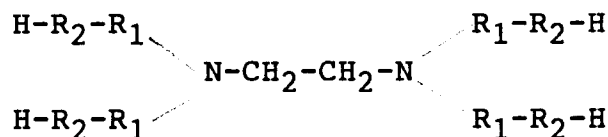
(11) Publication number:

0 465 000 A1

(12)

EUROPEAN PATENT APPLICATION(21) Application number: **91304645.4**(51) Int. Cl.⁵: **D21C 5/02, D21H 21/02**(22) Date of filing: **22.05.91**(30) Priority: **20.06.90 US 541240**(43) Date of publication of application:
08.01.92 Bulletin 92/02(84) Designated Contracting States:
AT BE DE ES FR GB IT NL SE(71) Applicant: **BETZ EUROPE, INC.**
4636 Somerton Road
Trevoze Pennsylvania 19047(US)(72) Inventor: **Pease, Jacqueline Kay**
12757 Ginger Drive
Jacksonville, FL.32223(US)
Inventor: **Dixon, Catherine Sangillo**
3330 Mush Bluff Road
St. Mary's GA. 31558(US)
Inventor: **Michalopoulos, Daniel Laurence**
4226 Chippendale Circle South
Jacksonville, FL.32217(US)(74) Representative: **W.P. Thompson & Co.**
Coopers Building, Church Street
Liverpool L1 3AB(GB)(54) **Composition for treating coated broke and its use.**

(57) A composition and method for inhibiting the deposition of white pitch on the surfaces of papermaking equipment during the processing of recycled coated broke. The process comprises adding to the coated broke during repulping the composition which comprises a first component which is a tetrafunctional alkoxyated diamine having the general formula:



wherein R₁ and R₂ are ethylene oxide or propylene oxide and R₁ and R₂ are not the same, and a second component selected from a phosphate, and a phosphonic acid or salt thereof.

EP 0 465 000 A1

The present invention relates to the paper processing industry and specifically addresses the problems caused by the generation of white pitch associated with the repulping of coated broke and other secondary fibres.

The problem of white pitch deposition occurs when secondary or coated paper is recycled. The coating, which comprises latex, generally styrene butadiene (SBR) and/or polyvinyl acetate (PVAC), natural binders, such as starch, and inorganic pigments, forms deposits on the paper machine. These deposits, referred to as "white pitch", can be very costly as they cause sheet defects, such as holes and breaks in the web of paper, as well as requiring added time for equipment cleanup. In the wet-end of the paper machine, white pitch usually deposits on the foils, table rolls, vacuum boxes, uhle boxes and throughout the press felts. Dry-end deposits appear on the dryer cans and dryer felts as a hazy white buildup.

US-A- 4 735 682 (Didwania et al) teaches a process for recovering papermaking fibres from latex-bonded broke or paper containing latex. The method described subjected the broke to an aqueous alkaline solution having a sodium hydroxide concentration in the range of 0.5 to 5 mole percent. This addition was used to eliminate clumps of fibres after pulping so that tissue could be made from the recovered cellulose.

Another method for removing and dispersing contaminants from secondary fibre during repulping is taught by US-A- 4 643 800 (Maloney et al). That method utilized an oxyethylene glycol nonionic surfactant in which one end hydroxyl group had been substituted with an aliphatic or alkylaromatic group and the other end hydroxyl group had been replaced with a polyoxypropylene group or a benzyl ether group. the nonionic surfactant was added in combination with a low molecular weight (500-50,000) polyelectrolyte dispersant.

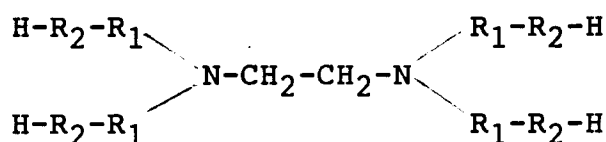
US-A- 3 808 089 (Von Koeppen et al) teaches a method for reuse of printed and coated paper as top liner. The method described utilized a combination of an ethoxylated aliphatic alcohol emulsifier with an alkali metal phosphate salt or silicate salt. The preferred ethoxylated aliphatic alcohols in that method were secondary alcohol ethoxylates.

The propensity for white pitch particles to deposit on papermaking equipment is related to the size of these particles. There is a direct link between white pitch deposition and the efficiency of breaking up larger coating particles into smaller ones during the repulping of recycled coated paper containing a latex coating.

There has been observed the effect of a change in the efficiency of breaking up the latex coating during repulping at a paper mill manufacturing medium weight coated publication grade paper. Prior to a change made to their coating formulation the mill experienced no problems from white pitch deposition. The change caused a significant increase in the particle size of the undispersed coating which remained even after repulping the coated paper for recycling purposes. A severe white pitch outbreak occurred as a result.

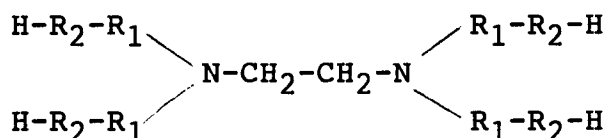
It is the function of the repulper to mechanically break down the larger coating particles into smaller ones. However, variances in the latex coating formulations between different papers result in variations in the sizes of the coating components after similar mechanical repulping operations. Some coating formulations will exhibit small coating particle sizes after repulping, while others, such as in the above noted mill example, will exhibit large particles after repulping. In order to prevent white pitch deposition and white pitch related sheet defects after the repulping process, these large particles must be reduced in size. Since the mechanical repulping operation is incapable of performing this task in every case where white pitch deposition or sheet spots and defects related to white pitch are a problem, a chemical additive is needed.

According to the present invention there is provided a composition for treating coated broke in a papermill having a repulping operation which comprises a first component which is a tetrafunctional alkoxyated diamine having the general formula:



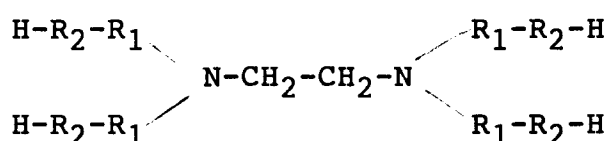
wherein R_1 and R_2 are ethylene oxide or propylene oxide and R_1 and R_2 are not the same, and a second component selected from a phosphate and a phosphonic acid or salt thereof.

The present invention also provides a process for minimizing the deposition of white pitch on papermaking equipment so as to reduce paper sheet defects, which comprises treating coated broke in a papermill having a repulping operation by adding to the coated broke a composition which comprises a first component which is a tetrafunctional alkoxyated diamine having the general formula:



wherein R_1 and R_2 are ethylene oxide or propylene oxide and R_1 and R_2 are not the same, and a second component selected from a phosphate and a phosphonic acid or salt thereof.

The present invention relates to a process for inhibiting the deposition of white pitch throughout a papermaking machine by breaking down the coating during repulping into smaller particles having a lower deposition potential. The process comprises adding to a repulper containing coated broke and water a combination of two chemical components. The first component is a tetrafunctional alkoxyated diamine having the following structure:



wherein R_1 and R_2 are ethylene oxide or propylene oxide and R_1 and R_2 are not the same. The ethylene oxide content in the molecule is preferably from about 10% to about 80% by weight, more preferably from about 10% to about 20% by weight. This molecule is available under the commercial product name Tetric from BASF Corporation. The second component is an anionic compound which contains a phosphate or phosphonate functionality. The two chemical components may be added to the repulper wherein the weight ratio of the tetrafunctional alkoxyated diamine to the phosphate or phosphonic acid and its salts is preferably from about 1:4 to 4:1. The more preferred ratio is about 1:1.

Since the processing of secondary fibres through a paper mill often results in the generation of problems and paper defects other than white pitch, it is expected that the present invention may be utilized in conjunction with items such as retention aids, felt conditioners and pitch control agents (when the coated broke is blended with virgin fibre).

Characteristic examples of the phosphate and phosphonic acid components are shown below:

$\text{Na}_5\text{P}_3\text{O}_{10}$: Sodium tripolyphosphate (STPP), FMC Corp.;

$(\text{NaPO}_3)_{21}$: glassy condensed phosphate (glass H) FMC Corp.;

amino trimethylene phosphonic acid (AMP), available from Monsanto Corp., as Dequest 2000;

1-hydroxyethylidene 1,1-diphosphonic acid (HEDP), available from Monsanto Corp., as Dequest 2010;

ethylene diamine tetramethylene phosphonic acid (EDTMP), available from Monsanto Corp., as Dequest 2041;

poly isopropenyl phosphonic acid (PIPA), available from Betz Laboratories, Inc;

polyoxyethylene nonyl phenyl ether phosphate, available from GAF Corp., as Gafac RE610;

polyoxyethylene octadecenyl ether phosphate, available from GAF Corp., as Gafac RB400;

amino trimethylene phosphonate;

hexamethylene diamine tetramethylene phosphonate;

ethylene diamine tetramethylene phosphonate;

diethylene triamine pentamethylene phosphonate.

The composition of the present invention is usually added to the coated broke within the repulping operation.

The coated broke usually contains one or more constituents selected from styrene butadiene and polyvinyl acetate.

The present invention will now be further described with reference to, but in no manner limited to, the following Examples.

Test 1:

Coated publication grade paper from a northern paper company referred to as "Coated Paper No.1" was used for the first study. This coated paper, which is produced in an alkaline environment, has an organic extractables content of 0.6%. The major extractable components are SBR and fatty acids, with a

minor component which is an organic ester. Coated Paper No.1 was pulped in a laboratory repulper at 5% consistency (percent filterable solids in water) in deionized water for 30 minutes at 38° C (100° F) and 1000 rpm; 5 minutes were allowed for warm-up to 38° C (100° F) prior to pulping. The desired chemical treatments were added directly to the pulper prior to warm-up. These treatments are listed as a percentage by weight of the total pulper contents. After repulping, the coated broke was diluted to 0.5% consistency and 3 to 4 microscope slides were made. The slides were evaluated for particles of undispersed coating which are easily distinguished from papermaking fibre. The particles were measured with an eyepiece micrometer scale which had been standardized to measure particles from 10 to 500 microns at 100x. Three passes were made across each slide and the particles were counted which fell within the following ranges: 100-200, 200-300, and greater than 300 microns. Particles less than 100 microns were not counted due to their relatively high number. The percent of the counted particles (those greater than 100 microns) which fell into each category was calculated and the average of the 3 to 4 slides was determined. Total value equals 100% + 2% due to overlap in computed averages. The results of this evaluation with no chemical additives are shown in Table I.

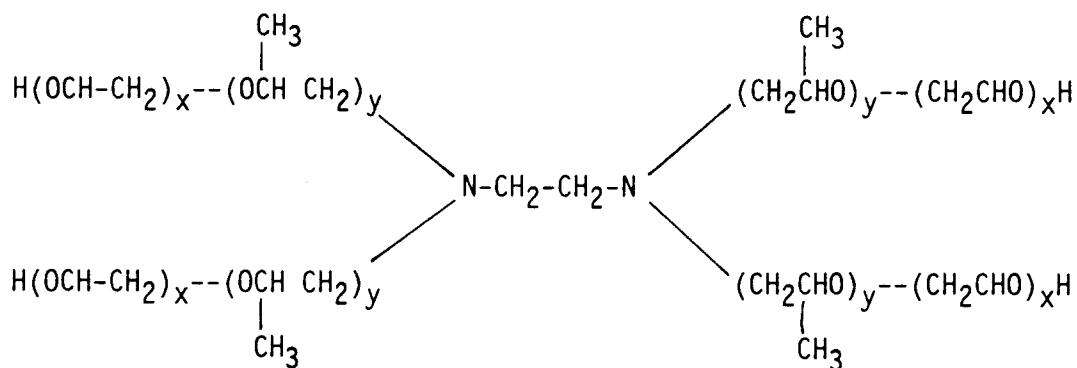
TABLE I
Coated Paper No.1
Percent of Particles Greater than 100 Microns

	<u>100-200</u>	<u>200-300</u>	<u>300 MICRONS AND GREATER</u>
No Additives	80%	18%	2%

With no additive, 20% of the counted particles of undispersed coating were larger than 200 microns. Particles this large are easily seen with the naked eye; appearing similar in size to sand granules. The results in Table III were obtained for Coated Paper No.1 when pulped in a conventional laboratory pulper, similar to a commercial papermill repulper with Na₅P₃O₁₀ (sodium tripolyphosphate) and a tetrafunctional diamine, added either separately or in combination. Table II defines and labels the tetrafunctional diamines which were utilized in the analysis of the invention and which are shown in the results provided herein.

TABLE II
Tetrafunctional Diamines

Example A1



with 10% by weight of x, ethylene oxide.

Example A2

same as formula in A1, except with 20% by weight of x, ethylene oxide.

Example A3

same as formula in A1, except with 40% by weight of x, ethylene oxide.

Example A4

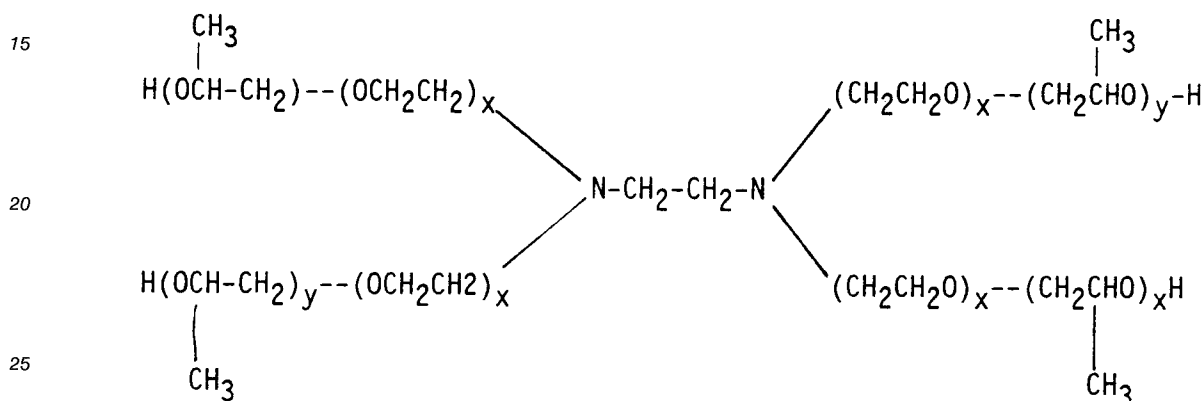
5 same as formula in A3, except having a higher molecular weight.

Example A5

same as formula in A1, except with 80% by weight of x, ethylene oxide.

10

Example B1



with 40% by weight of x, ethylene oxide.

30

Example B2

same as formula in B1, except with 10% by weight of x, ethylene oxide.

Example B3

same as formula in B1, except with 20% by weight of x, ethylene oxide.

40

TABLE III
Tetrafunctional diamine/phosphate efficacy

<u>Composition</u>	<u>Concentration percent by weight</u>	<u>Particle Size After Treatment (Microns)</u>		
		<u>100-200</u>	<u>200-300</u>	<u>300+</u>
45 $\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.025 .025	98%	2%	0
Example A1	.05	93%	7%	0
50 $\text{Na}_5\text{P}_3\text{O}_{10}$	.05	88%	12%	0

55

A test was performed to determine the likelihood that the broke will stick to papermaking equipment surfaces, such as the dryer can, and cause deposition related problems. Paper sheets were made using a conventional Noble and Wood sheet mold from pulped Coated Paper No.1. Foil was attached and dried to the sheets and the force required to peel the foil from the paper was measured. The higher the amount of peel strength the more likely the broke will stick to the dryer can. These data measuring increasing dosage levels are shown in Table IV.

TABLE IV
Peel Strength

	<u>Composition</u>	<u>Concentration Percent by Weight</u>	<u>Peel strength (g/cm width)</u>
5	No Additives		30.0
10	$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.0125 .0125	25.8
	$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.025 .025	24.5
15	$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.05 .05	23.9

These results correspond with the field and laboratory data shown in Tables I and III. It was observed that as the percent of larger coating particles was reduced by the additives of the present invention, the deposition potential of the paper made with the recycled coated broke decreased.

Other phosphates were tested for their efficacies when combined with the tetrafunctional diamine, Example A1. Results were obtained in accordance with the laboratory test procedure defined hereinabove and are shown in Table V.

TABLE V
Alternative phosphates/tetrafunctional diamine efficacy

	<u>Composition</u>	<u>Concentration percent by weight</u>	<u>Particle Size After Treatment (Microns)</u>		
			<u>100-200</u>	<u>200-300</u>	<u>300+</u>
30	NaH_2PO_4 &	.025			
35	Example A1	.025	97%	3%	0%
	NaH_2PO_4	.05	84	16	0
40	$(\text{NaPO}_3)_{21}$ &	.025			
	Example A1	.025	99%	1%	0
45	$(\text{NaPO}_3)_{21}$	.05	85	15	0

The above results indicate that other phosphates may be successfully combined with the tetrafunctional diamine to produce an effective additive for reducing particle size. It also appears that the higher the molecular weight the better the results as the larger molecular weight compound $(\text{NaPO}_3)_{21}$, combined with the tetrafunctional diamine to yield only 1% of particles being 200 microns or greater in size. Accordingly, other phosphates may be effectively utilized with the tetrafunctional diamine. These include ortho-phosphates such as, for example, monopotassium phosphate, polyphosphates such as, for example, tetra-potassium pyrophosphate and glassy phosphates such as, for example, hexametaphosphate.

Other tetrafunctional diamines, differing from each other in relative amounts of ethylene oxide, were pulped with Coated Paper No.1 in combination with $\text{Na}_5\text{P}_3\text{O}_{10}$. Due to lack of experimental significance, where the combination selected showed little or no beneficial effect, that particular tetrafunctional diamine was not pulped at an equal dosage level independently. These results are shown in Table VI.

TABLE VI
Alternative tetrafunctional diamines/ $\text{Na}_5\text{P}_3\text{O}_{10}$ efficacies

Composition	Concentration percent by weight	Particle Size (Microns)		
		100-200	200-300	300+
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A5	.025 .025	86%	12%	1%

TABLE VI (Cont'd)

Alternative tetrafunctional diamines/ $\text{Na}_5\text{P}_3\text{O}_{10}$ efficacies				
Composition	Concentration percent by weight	Particle Size (Microns)		
		100-200	200-300	300+
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A4	.025 .025	89%	10%	1%
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A3	.025 .025	89	8	3
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example B1	.025 .025	89	11	0
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A2	.025 .025	99	1	0
Example A2	.05	94	5	1
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example B3	.025 .025	97	3	0
Example B3	.05	84	15	2
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example B2	.025 .025	96	4	0
Example B2	.05	91	9	0

The data from Table VI indicates that not all tetrafunctional diamines are effective when added in combination with $\text{Na}_5\text{P}_3\text{O}_{10}$. 10 to 20% ethylene oxide content provides the most preferred efficacy.

Table VII, below, shows data from tests conducted using other nonionic compounds in place of the tetrafunctional diamine.

TABLE VII

Efficacy of alternative nonionic compounds

Composition	Concentration percent by weight	Particle Size (Microns)		
		100-200	200-300	300+
Na ₅ P ₃ O ₁₀ & Example C1	.025 .025	80%	19%	1%
Na ₅ P ₃ O ₁₀ & Example C2	.025 .025	82	15	3
Na ₅ P ₃ O ₁₀ & Example C3	.025 .025	89	11	1
Example C3	.05	88	10	2
Na ₅ P ₃ O ₁₀ & Example C4	.025 .025	86	14	1
Example C4	.05	86	12	1
Na ₅ P ₃ O ₁₀ & Example C5	.025 .025	93	6	1
Example C5	.05	96	4	1
Example C1)	ethyleneoxy propyleneoxy block copolymer, 30% by weight ethylene oxide.			
Example C2)	ethyleneoxy propyleneoxy block copolymer 80% by weight ethylene oxide.			
Example C3)	ethyleneoxy propyleneoxy block copolymer 10% by weight ethylene oxide.			
Example C4)	butoxy polypropyleneoxy polyethyleneoxy ethanol.			
Example C5)	Secondary alcohol ethoxylate.			

The alternative nonionic compounds of Table VII also contain repeat units of ethylene oxide and propylene oxide. However, although similar to the tetrafunctional diamines, they do not perform in the same manner when combined with phosphate.

The tetrafunctional diamine component of the present invention may be effectively utilized with anionic species other than the phosphate shown in the test results above. These data are shown in Table VIII.

TABLE VIII

Alternative Anionics/Tetrafunctional Diamine Efficacy

Composition	Concentration percent by weight	Particle Size (Microns)		
		100-200	200-300	300+
Example D1 & Example A1	.025 .025	91%	8%	1%
Example D2 & Example A1	.025 .025	99	1	0
Example D2	.05	92	8	1
Example D3 & Example A1	.025 .025	96	4	0
Example D3	.05	89	10	1
Example D4 & Example A1	.025 .025	87	12	1
Example D4	.05	79	18	2
Example D1)	ethylene diamine tetra acetic acid			
Example D2)	hydroxyethylidene diphosphonate			
Example D3)	sodium salt of polyisopropenyl phosphonic acid			
Example D4)	sodium salt of sulfonated naphthalene - formaldehyde condensate			

The products which contain a phosphate functionality, hydroxyethylidene diphosphonate and the sodium salt of polyisopropenyl phosphonic acid, performed exceptionally well in combination with the tetrafunctional diamine. Accordingly, other phosphonates may be effectively utilized in accordance with this invention. Characteristic examples include amino trimethylene phosphonate, hexamethylene diamine tetramethylene phosphonate, ethylene diamine tetramethylene phosphonate and diethylene triamine pentamethylene phosphonate.

Test 2

Coated publication paper from another paper manufacturer was used in these studies and will be referred to as "Coated Paper No.2". This paper is made by an acid papermaking process. Coated Paper No.2 contains a higher level of organic extractables (2.3%) than Coated Paper No.1. These extractables contain a major component of SBR and minor components of PVAC, organic acids, and organic acid salts. The studies with this broke were carried out in the same manner as those in Example 1 except the particles were counted in slightly different particle size ranges (100-150, 150-200, 200-300, and 300 microns and greater). Once again particles less than 100 microns were not counted due to their high numbers.

TABLE IX

Coated Paper No.2
Percent of Particles Greater than 100 Microns

<u>Compound</u>	<u>100-150</u>	<u>100-200</u>	<u>200-300</u>	<u>300+</u>
No Additives	77%	15%	6%	2%

Table X shows the effects of adding $\text{Na}_5\text{P}_3\text{O}_{10}$ and the tetrafunctional diamine of Example A1 either independently or in combination during the pulping of Coated Paper No.2.

TABLE X

$\text{Na}_5\text{P}_3\text{O}_{10}$ /Tetrafunctional Diamine Efficacy

<u>Compound</u>	<u>Concentration</u>	<u>Particle size (Microns)</u>			
	<u>Percent by Weight</u>	<u>100-150</u>	<u>150-200</u>	<u>200-300</u>	<u>300+</u>
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.025				
	.025	90%	9%	1%	0%
$\text{Na}_5\text{P}_3\text{O}_{10}$	.05	80	17	3	0
Example A1	.05	80	12	9	0

Even though this coated broke is different than Coated Paper No.1, in both cases the combination of $\text{Na}_5\text{P}_3\text{O}_{10}$ and the tetrafunctional diamine, Example A1, produced fewer large particles than when each component was added separately.

As in Example No.1, paper was made from Coated Paper No.2 which had been repulped with increasing quantities of the $\text{Na}_5\text{P}_3\text{O}_{10}$ /Example A1 combination. The amount of force required to separate foil and paper, which had been dried together, was measured and is indicative of the deposition potential in the dryer section of the paper machine (the higher the peel force the greater the deposition potential). The results are presented in Table XI.

TABLE XI
Peel Strength

	<u>Compound</u>	<u>Concentration Percent by Weight</u>	<u>Peel Strength (g/cm width)</u>
5	No Additives	-	22.6
10	$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.00625 .00625	19.9
15	$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example 1	.0125 .0125	18.1
20	$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.025 .025	15.5
25	$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example A1	.05 .05	13.9

As also seen in Example No.1, increasing dosages of the tetrafunctional diamine/ $\text{Na}_5\text{P}_3\text{O}_{10}$ combination caused a proportional continuing decrease in the deposition potential of the repulped Coated Pair No.2 as measured by peel strength.

Coated Paper No.2 was pulped with the tetrafunctional diamine Example A1 and various phosphonates. The results are shown in Table XII.

TABLE XII
Phosphonates/Tetrafunctional Diamine Efficacy

5	<u>Compound</u>	<u>Concentration</u> <u>Percent by Weight</u>	<u>Particle size (Microns)</u>			
			<u>100-150</u>	<u>150-200</u>	<u>200-300</u>	<u>300+</u>
	Example E1 & Example A1	.025 .025	-- too few particles greater than 100 microns -			
10	Example E2 & Example A1	.025 .025	-- too few particles greater than 100 microns			
15	Example E3 & Example A1	.025 .025	-- too few particles greater than 100 microns -			
	Example E2 & Example A1	.00625 .00625	89%	8%	4%	0%
20	Example A1	.0125	80	13	4	2
	Example E2	.0125	83	12	4	2
25	Example E3 & Example A1	.00625 .00625	89	6	4	2
	Example E3	.0125	84	14	2	1
30	Example E1	amino trimethylene phosphonic acid				
	Example E2	1-hydroxyethylidene 1,1-diphosphonic acid				
	Example E3	ethylene diamine tetramethylene phosphonic acid				

35 The phosphonate/tetrafunctional diamine combinations proved to be much more effective than the Na₅P₃O₁₀/tetrafunctional diamine combinations on reducing particle size in the pulped Coated Paper No.2. In fact, the treatment concentration level needed to be reduced in order find a sufficient number of particles larger than 100 microns.

40 The deposition potential of Coated Paper No.2 when repulped with 1 hydroxyethylidene 1,1-diphosphonic acid/tetrafunctional diamine was evaluated in accordance with the procedure previously described. Results are shown in Table XIII.

45

50

55

TABLE XIII
Peel Strength

	<u>Compound</u>	<u>Concentration Percent by Weight</u>	<u>Peel Strength (g/cm width)</u>
5	No Additives	-	25.7
10	Example E2 & Example A1	.0025 .0025	24.3
15	Example E2 & Example A1	.00625 .00625	21.0
	Example E2 & Example A1	.0125 .0125	17.5
20	Example E2 & Example A1	.025 .025	14.1

The deposition potential of Coated Paper No.2 decreased with increasing concentrations of the phosphonate/tetrafunctional diamine compound. This correlates with a corresponding particle size reduction with increasing concentrations of the compound.

Other anionic species were tested for particle size reduction efficacy in Coated Paper No.2 along with the tetrafunctional diamine. These results are shown in Table XIV.

TABLE XIV
Alternative Anionic/Tetrafunctional Diamine Efficacy

Compound	Concentration Percent by Weight	Particle size (Microns)			
		100-150	150-200	200-300	300+
Example F1 & Example A1	.025 .025	82%	11%	5%	1%
Example F1	.05	83	11	2	4
Example F2 & Example A1	.025 .025	92	7	1	0
Example F2	.05	84	10	4	1
Example F3 & Example A1	.025 .025	83	14	3	0
Example F3	.05	82	13	4	1
Example F4 & Example A1	.025 .025	79	18	2	2
Example F4	.05	75	18	5	2
Example F1)	polyacrylic acid				
Example F2)	polyisopropenyl phosphonic acid				
Example F3)	polyoxyethylene nonyl phenyl phosphate ester				
Example F4)	polyoxyethylene octadecenyl phosphate ester				

The only effective combination shown above was the tetrafunctional diamine with the phosphonic acid, Example F2. These results correspond with the testing conducted on Coated Paper No.1. The cumulative data from Example 1 and Example 2 support the finding that any phosphate functionality will act synergistically with tetrafunctional diamines to effectively redisperse the latex coating in the smallest particles possible during repulping of recycled coated paper.

As in Example 1, $\text{Na}_5\text{P}_3\text{O}_{10}$ was combined with other nonionic surfactants. The results are shown in Table XV.

TABLE XV
 $\text{Na}_5\text{P}_3\text{O}_{10}$ /Alternate Nonionic Surfactant Efficacy

Compound	Concentration Percent by Weight	Particle size (Microns)			
		100-150	150-200	200-300	300+
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example C5	.025 .025	 88%	 10%	 2%	 0%
Example C5	.05	90	9	1	1
$\text{Na}_5\text{P}_3\text{O}_{10}$ & Example C3	.025 .025	 90	 6	 2	 1
Example C3	.05	82	10	4	4

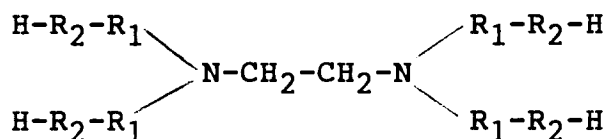
Example C5) secondary alcohol ethoxylate

Example C3) ethyleneoxy propyleneoxy block copolymer,
 with 10% by weight of ethylene oxide.

Neither the alcohol ethoxylate nor the block copolymer performed effectively with the phosphate to reduce particle size. These results correlate with findings under Example 1.

Claims

1. A composition for treating coated broke in a papermill having a repulping operation which comprises a first component which is a tetrafunctional alkoxyated diamine having the general fomrula:

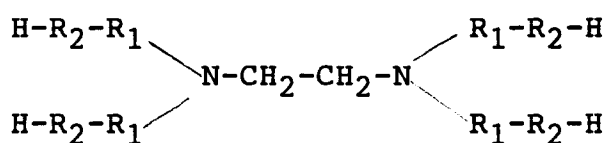


wherein R_1 and R_2 are ethylene oxide or propylene oxide and R_1 and R_2 are not the same, and a second component selected from a phosphate and a phosphonic acid or salt thereof.

2. A composition according to claim 1, wherein the ethylene oxide content of the tetrafunctional alkoxyated diamine is from about 10% to about 80% by weight.
3. A composition according to claim 2, wherein the ethylene oxide content of the tetrafunctional alkoxyated diamine is from about 10% to about 20% by weight.
4. A composition according to any of claims 1 to 3 wherein the phosphate is selected from $\text{Na}_5\text{P}_3\text{O}_{10}$, $(\text{NaPO}_3)_{21}$, polyoxyethylene nonyl phenyl ether phosphate, and polyoxyethylene octadecenyl ether phosphate.
5. A composition according to any of claims 1 to 3, wherein the phosphonic acid or salt thereof is selected

from amino-trimethylene phosphonate, hexamethylene diamine tetramethylene phosphonate, ethylene diamine tetramethylene phosphonate diethylene triamine penta methylene phosphonate, 1-hydroxyethylidene 1,1-di-phosphonic acid, ethylene diamine tetramethylene phosphonic acid and polyisopropenyl phosphonic acid.

- 5
6. A composition according to any of claims 1 to 5, wherein the weight ratio of the tetrafunctional alkoxyated diamine to the second component is from about 1:4 to about 4:1.
7. A composition according to claim 6 wherein the weight ratio is about 1:1.
- 10
8. A process for minimizing the deposition of white pitch on papermaking equipment so as to reduce paper sheet defects, which comprises treating coated broke in a papermill having a repulping operation by adding to the coated broke a composition which comprises a first component which is a tetrafunctional alkoxyated diamine having the general formula:



wherein R_1 and R_2 are ethylene oxide or propylene oxide and R_1 and R_2 are not the same, and a second component selected from a phosphate and a phosphonic acid or salt thereof.

- 25
9. A process according to claim 8, wherein the first component and/or second component are as defined in any of claims 1 to 7.
10. A method according to claim 9, wherein the coated broke contains one or more constituents selected from styrene butadiene and polyvinyl acetate.
- 30
11. A method according to any of claims 8 to 10 wherein the composition is added to the coated broke within the repulping operation.
- 35
12. A method according to any of claims 8 to 11, wherein the composition is utilized in conjunction with one or more additive(s) selected from retention aids, felt conditioners and pitch control agents.



European
Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91 30 4645

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	EP-A-0 232 015 (W. R. GRACE AB) * page 2, line 5 - line 8 *** page 4, line 9 - line 10; claims ** - - -	1,2,8,11, 12	D 21 C 5/02 D 21 H 21/02
A	US-A-765 867 (BETZ LABORATORIES INC.) * column 3, line 43 - line 58; claims ** - - -	1,8,12	
A	FR-A-926 742 (NATIONAL OIL PRODUCTS) * page 1, line 50 - page 2, line 56 ** - - - - -	1,8,11	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			D 21 C D 21 H
The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of search 08 October 91	Examiner BERNARDO NORIEGA F.
CATEGORY OF CITED DOCUMENTS X: particularly relevant if taken alone Y: particularly relevant if combined with another document of the same category A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention E: earlier patent document, but published on, or after the filing date D: document cited in the application L: document cited for other reasons ----- &: member of the same patent family, corresponding document			